Monsanto

800 N. Lindbergh Boulevard St. Louis, Missouri 63166 (314) 694-1000

algues to Office

(314) 694-1000	SALES AGREEMENT
	(Industrial Materials)
SOLD TO	Western Forest Products Limited ("Buyer")
	1111 West Georgia Street
	Vancouver, British Columbia, Canada , the following
industrial ma	terial (Material''), described generally in Exhibit A attached hereto and made a part hereof, as generated from time to
time at Monsa	anto'sWashington
plant, located	at or near Seattle
("Plant"), sub	ject to the terms and conditions as stated below and on pages 2 and 3 of this Agreement.
provided that be confirmed action or required Buyer is hand in the course this Agreemen	2
Monsanto, sub Monsanto sha materials the Agreement be which Monsar operations wh	ITY. Buyer agrees to accept delivery from Monsanto attementary of such quantities of the Material as are offered by oject to any minimum and maximum as may be set forth in Exhibit A. Anything herein to the contrary notwithstanding, all not be required to operate any part of any of its manufacturing facilities or to purchase from other sources any same as or similar or dissimilar to the Material for the purpose of supplying Material to Buyer, the purpose of this ing to sell a material which may be incidentally produced by Monsanto in the course of its operation of the Plant and into may choose to offer to Buyer. Buyer acknowledges that Monsanto may at any time reduce or cease production nich produce the Material and thereby reduce or discontinue the available supply of the Material and agrees that all led or not made as a result thereof shall be so reduced or cancelled without liability.
shall be due w terminate this b. Any tax (i) increases to production, sa tax, may at M c. The price	**RAXMSX RECENTED A: a. Buyer shall pay Monsanto for the Material at the rate set forth in Exhibit A. Payment vithin thirty (30) days from the date of Monsanto's invoice. Monsanto reserves the right, among other remedies, either to Agreement or to suspend further deliveries under it in the event Buyer fails to make any payment when due. or governmental charge or increase in same (excluding any franchise or income tax or other tax based on income) which he cost to Monsanto of producing, selling or delivering the Material or which (ii) is payable by Monsanto because of the ale or delivery of the Material, such as sales tax, use tax, retailer's occupational tax, gross receipts tax and value added consanto's option be added to the price herein specified. The production of delivery, and terms of payment herein specified may be revised by Monsanto at any time upon giving Buyer at lired (100) days' prior notice thereof.
writing, of the as scheduled by accumulate in and cause its e b. Buyer, a	ERY; TITLE AND RISK OF LOSS. a Monsanto shall give Buyer at least five (5) days' advance notice, orally or in initial availability of the Material. Following receipt of such notice, Buyer shall take delivery of the Material at the Plant by Monsanto, between the hours of 8:00 a.m. and 4:00 p.m., Monday through Friday except holidays, unless otherwise Monsanto, and in a manner not inconvenient to the operation of the Plant. Buyer shall not permit the Material to quantities requiring storage or creating inconvenience to Monsanto's operation of the Plant. Buyer shall comply with, employes and agents to comply with, the Plant's safety and security rules while at the Plant. It its expense, shall supply or arrange for the necessary transportation equipment and for the transportation required to erial safely and lawfully from the Plant to Buyer's destination.
	v 022254
	(TERMS AND CONDITIONS CONTINUED ON PAGES 2 AND 3 HEREOF) nt shall not be binding on Monsanto unless executed by Buyer and an authorized representative of Monsanto and onsanto within thirty days from the date below.
EXECUTED BY	West Maruets Smitch MONSANTO COMPANY 3/3/82
BY /	en turchasing THE

TERMS AND CONDITIONS (continued)

- - d. Monsanto's weights shall govern unless proved to be in error by more than one percent (1%).
 - 5. WARNING, CLAIMS AND LIMITED LIABILITY. a. A GENERAL DESCRIPTION OF THE MATERIAL IS SET FORTH IN EXHIBIT A BUT SUCH DESCRIPTION DOES NOT CONSTITUTE ANY WARRANTY OR REPRESENTATION THAT THE MATERIAL SHIPPED WILL CONFORM TO SUCH DESCRIPTION OR THAT SUCH DESCRIPTION IS COMPLETE OR ACCURATE. THE COMPOSITION OF THE MATERIAL IS EXPECTED TO VARY FROM SHIPMENT TO SHIPMENT AND MONSANTO THEREFORE SELLS THE MATERIAL TO BUYER ON AN "AS IS" BASIS, "WITH ALL FAULTS," AND WITHOUT ANY EXPRESS OR IMPLIED WARRANTY.
 - b. Buyer acknowledges and agrees that the Material will vary from time to time in composition and other characteristics. Buyer assumes the risk of such variances and the risk of evaluating and ascertaining, and will examine, evaluate and ascertain, within thirty (30) days after receipt of each shipment, the quality and condition of the Material as well as any health, safety or environmental hazards related thereto. All claims by Buyer for any cause whatsoever (whether based in contract, warranty, negligence, strict liability, other tort, violation of law, or otherwise) shall be deemed waived unless made in writing and received by Monsanto within forty-five (45) days after delivery to Buyer of the Material in respect to which such claim is made. IN THE EVENT MONSANTO SHALL INCUR OR BE SUBJECT TO ANY LIABILITY WITH RESPECT TO THIS AGREEMENT OR THE MATERIAL, BUYER'S EXCLUSIVE REMEDY SHALL BE FOR DAMAGES, AND MONSANTO'S TOTAL LIABILITY FOR ANY AND ALL LOSSES AND DAMAGES ARISING OUT OF ANY CAUSE WHATSOEVER (WHETHER SUCH CAUSE BE BASED IN CONTRACT, WARRANTY, NEGLIGENCE, STRICT LIABILITY, OTHER TORT, VIOLATION OF LAW OR OTHERWISE) SHALL IN NO EVENT EXCEED THE PUR-CHASE PRICE OF THE MATERIAL IN RESPECT TO WHICH SUCH CAUSE ARISES OR, AT MONSANTO'S OPTION, THE REPLACEMENT OF SUCH MATERIAL. IN NO EVENT SHALL MONSANTO BE LIABLE FOR INCIDENTAL, SPECIAL, CON-SEQUENTIAL OR PUNITIVE DAMAGES RESULTING FROM ANY SUCH CAUSE, THIS PARAGRAPH SHALL IN NO WAY CREATE OR IMPLY ANY LIABILITY TO MONSANTO NOR DIMINISH ANY OF MONSANTO'S RIGHTS UNDER SECTION 6, INDEMNIFICATION, OF THIS AGREEMENT.
 - If, due to a permanent change in composition of the Material because of a change in feedstocks or raw materials used by Monsanto or an alteration of Monsanto's method of manufacture, Buyer is no longer able to use the Material, Buyer may, upon giving Monsanto at least thirty (30) days' prior notice, terminate this Agreement without liability. Monsanto shall in no event be liable for damages to Buyer resulting from such termination or the events causing such termination.
 - c. If Monsanto furnishes technical or other advice to Buyer, whether or not at Buyer's request, Buyer shall verify the accuracy and application thereof prior to its use or implementation, and Monsanto shall not be liable for, and Buyer assumes all risk of, such advice and the results thereof. No inspection or review, if any, by Monsanto of Buyer's facilities or operations shall relieve Buyer of any of its obligations under this Agreement.
 - 6. INDEMNIFICATION. Buyer assumes sole responsibility for, and shall indemnify and hold harmless Monsanto, its present, past and future employes and agents, from and against any and all claims, liabilities, suits, proceedings, judgments, orders, fines, penalties, damages, losses, costs and expenses (including, without

limitation, costs of defense, settlement and reasonable attorneys' fees and expenses), (all of the foregoing herein collectively called "Liabilities, Proceedings and Damages"), arising out of or connected with (a) the purchase, receipt, delivery, transportation, handling, storage, treatment, processing, consumption, possession, use, resale or disposition by or on behalf of Buyer, of any Material or any component thereof, whether the Material or any component thereof is used alone or in combination with any other material; or (b) any failure of Buyer or any of its employes or agents to observe or comply with any of Buyer's duties or obligations under this Agreement, including, without limitation, any failure to observe or comply with any applicable laws, ordinances, codes, orders, rules or regulations. The foregoing obligations of Buyer shall include, but not be limited to, any and all Liabilities, Proceedings and Damages for or relating to (i) injury to or death of any person (including, without limitation, employes and agents of Buyer or Monsanto), (ii) damage to or loss or destruction of any property (including, without limitation, property of Buyer or Monsanto, or their employes and agents), and (iii) any contamination of, injury or damage to or adverse effect on persons, animals, aquatic and wild life, vegetation, waters, air, land or the environment.

The foregoing indemnification shall apply regardless of the basis of liability or legal principle involved (including, without limitation, contract, warranty, negligence, strict liability, other tort, violation of law or otherwise), but shall not apply to Liabilities, Proceedings and Damages resulting solely and directly from Monsanto's negligence or willful or criminal misconduct.

Upon the request of Monsanto or any other person or party covered by the foregoing indemnification, Buyer shall, at its expense, cause any such claims, liabilities, suits or proceedings to be defended by counsel approved by Monsanto, and Monsanto shall have the right, at Buyer's expense, to participate in the investigation, defense, settlement and/or compromise of same.

- 7. **INSURANCE BY BUYER**. a. Buyer shall not begin its performance under this Agreement until:
 - It has obtained all the insurance hereinafter required; and
 - (ii) It has furnished Monsanto with certificates of insurance satisfactory to Monsanto evidencing such required coverage; and
 - (iii) Copies of any provisions in Buyer's contract(s) of insurance excluding coverage for pollution have been provided to Monsanto.
- b. Every contract of insurance providing the coverages required herein shall provide that such coverages shall not be terminated, reduced or allowed to expire without the insurance carrier first giving Monsanto at least thirty (30) days' prior written notice thereof, and Buyer shall make such arrangements as are necessary to ensure that no termination, reduction or expiration of the insurance required herein becomes effective until thirty (30) days after Monsanto receives such notice.
- c. Buyer shall take out and maintain, at its expense, during the term of this Agreement, and for a minimum of two (2) years following the expiration or termination of this Agreement, at least the following insurance in insurance companies satisfactory to Monsanto:

	Coverage	L.1111115
(1)	Workmen's Compensation	Statutory
(2)	Employer's Liability	\$500,000 each occurrence
(3)	Public Liability	\$500,000 each person
	(Bodily Injury)	\$1,000,000 each occurrence
(4)	Public Liability	\$1,000,000 each occurrence
	(Property Damage)	
(5)	Automobile Liability	\$1,000,000 combined
	(Bodily Injury and	single limit
	Property Damage)	-

d. The insurance certificate evidencing the required coverage

TERMS AND CONDITIONS (continued)

shall include a certification that the above described insurance coverages include contractual coverage for the Buyer's liability under this Agreement.

- e. Buyer shall secure from the company carrying Buyer's Workmen's Compensation insurance a waiver of subrogation in favor of Monsanto and its employes and agents and shall furnish to Monsanto a copy of said waiver.
- f. The insurance requirements set forth herein are minimum coverage requirements and are not to be construed in any way as a limitation on Buyer's liability under this Agreement.
- 8. BUYER'S WARRANTIES AND COVENANTS. Buyer warrants to and covenants with Monsanto that:
- a. Buyer has the requisite experience, knowledge and expertise, suitable facilities, qualified personnel and legal right to receive, purchase, transport, handle, store, treat, process, use and dispose of the Material; and
- b. Buyer shall comply with all laws, ordinances, regulations, orders and actions of the United States and of any state, county, township or municipal subdivision or other governmental agency which may now or hereafter be applicable to the receipt, possession, transportation, placarding, handling, processing, storing, labeling, use, resale or disposition of the Material by or on behalf of Buyer; and
- c. Buyer has obtained and shall keep in effect all permits, licenses and other forms of documentation required now or hereafter in order to comply with such governmental orders, regulations, ordinances, actions or laws, and agrees to furnish copies of the same to Monsanto upon request without charge to Monsanto: and
- d. In processing, using and disposing of the Material, Buyer shall not use any Monsanto trademark without its prior written consent; and
- e. Buyer knows and understands that the Material is, or may have been in contact with or contain materials that are, flammable, toxic, corrosive, dangerous, hazardous or defective. Certain information as to the Material and certain recommended precautions for exposure to and handling of the Material are set forth or referred to in Exhibit A. Buyer agrees to advise and inform its employes, agents, representatives and subcontractors and any purchasers of the Material of the nature of the Material, the hazards associated with the Material, any recommended handling precautions and all appropriate measures to ensure the safety and well-being of persons, property and the environment prior to such individuals' commencement of work involving the Material or such purchase. Buyer agrees that it will promptly inform Monsanto in writing of any health, safety or environmental hazard relating to the Material which becomes known to Buyer subsequent to the date of this Agreement.
- 9. NOTIFICATION OF CITATIONS AND CLAIMS. Buyer agrees that it will promptly notify Monsanto of any of the following which relates to or is connected with Buyer's purchase, handling, hauling, storing, processing, use, resale or disposal of the Material: (i) any warning, citation, indictment, claim, lawsuit or proceeding issued or instituted by any federal, state or local governmental entity or agency against or upon Buyer; or (ii) the revocation of any license or permit issued to Buyer by any such entity or agency; or (iii) any other claim (including, without limitation, claims for Workmen's Compensation) or lawsuit against Buyer for personal injury, death or property damage.
- EXCUSE OF PERFORMANCE. The performance or observance by either party of any obligations of such party under this Agreement may be suspended by it, in whole or in part, and without liability in the event of any of the following which prevents such performance or observance: Act of God, war, riot, fire, explosion, accident, flood, sabotage, strike, lockout, injunction, inability to obtain fuel, power, raw materials, labor, containers of transportation facilities, breakage or failure of machinery or apparatus, national defense requirements, com-

- pliance with governmental laws, regulations, orders or action, or any other cause (whether similar or dissimilar) beyond the reasonable control of such party; provided, however, that the party so prevented from complying with its obligations hereunder shall immediately notify in writing the other party thereof and such party so prevented shall exercise diligence in an endeavor to remove or overcome the cause of such inability to comply, and provided further that neither party shall be required to settle a labor dispute against its own best judgment. Deliveries suspended or not made by reason of this section shall be cancelled without liability, but this Agreement shall otherwise remain unaffected. Nothing in this Section 10 shall excuse Buyer from performance or observance of its obligations under this Agreement by reason of its faiture or inability to observe or comply with Section 8 b. or of this Agreement.
- 11. **ASSIGNMENT.** Buyer may not, whether by operation of law or otherwise, assign, subcontract or otherwise transfer any of its rights nor delegate the performance of any of its obligations hereunder without Monsanto's prior written consent, and any attempted assignment, subcontracting, transfer or delegation without such consent shall be void and of no effect. Subject to the foregoing, this Agreement shall inure to the benefit of and be binding upon the parties hereto and their respective successors and assigns.
- 12. **NOTICES.** Except as otherwise provided in this Agreement, any notice, request, approval or other document required or permitted to be given under this Agreement shall be in writing and shall be deemed to have been sufficiently given when delivered in person or deposited in the U.S. mail, postage prepaid, addressed as specified in Exhibit A or to such other address or addresses as may be specified from time to time in a written notice given by such party. The parties shall acknowledge in writing the receipt of any such notice delivered in person.
- 13. MISCELLANEOUS. a. This Agreement constitutes the full understanding of the parties, a complete allocation of risks between them and a complete and exclusive statement of the terms and conditions of their agreement; and all prior agreements, negotiations, dealings and understandings, whether written or oral, regarding the subject matter hereof, are hereby superseded and merged into this Agreement. No conditions, usage of trade, course of dealing or performance, understanding or agreement purporting to modify, vary, explain or supplement the terms or conditions of this Agreement shall be binding unless hereafter made in writing and signed by the party to be bound, and no modification shall be effected by the acknowledgment or acceptance of purchase order or shipping instruction forms containing terms or conditions at variance with or in addition to those set forth in this Agreement. No waiver by either party with respect to any breach or default or of any right or remedy and no course of dealing or performance shall be deemed to constitute a continuing waiver of any other breach or default or of any other right or remedy, unless such waiver be expressed in writing signed by the party to be bound.
- b. As used in this Agreement, employes or agents of a party hereto shall be deemed to include, without limitation, such party's past, present and future officers and directors.
- c. Section headings as to the contents of particular sections are for convenience only and are in no way to be construed as part of this Agreement or as a limitation of the scope of the particular sections to which they refer.
- d. The validity, interpretation and performance of this Agreement and any dispute connected herewith shall be governed and construed in accordance with the laws of the State of Missouri.
- e. If any term or provision of this Agreement or any application thereof shall be invalid or unenforceable, the remainder of this Agreement or any other application of such term or provision shall not be affected thereby.

SEE EXHIBIT A ATTACHED.

EXHIBIT A

\		-74111271174		
	To Sales Agreement b	oetween Monsanto	Company and	
	Dated			
1. General Description of	the Material			
Components		Estimat	ed Range (%)	
2. Quantity				
2. <u>Quantity</u> (Min./Max. if any)				
3. <u>Price</u>				
4. Handling Precautions	<i>'</i>			
5. <u>Address for notices</u> Monsanto:		Buyer:	La La Mark	# 1 M
			TO THE REAL PROPERTY OF THE PARTY OF THE PAR	rilegista Dance

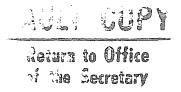


EXHIBIT A

To Sales Agreement between Monsanto Company and

Western Forest Products Limited

Dated	L

I. EXPLANATION OF CHANGES IN SALE AGREEMENT

- on March 1, 1982 and shall continue in full force and effect through February 28, 1985 and continue from year to year thereafter (March through February) unless and until terminated as of February 28, 1985 or as of the last day of any February thereafter by either party giving the other party at least six (6) months prior written notice of said termination.
- 2 Buyer's Mill, Woodfibre, British Columbia
- 3 unloaded at Buyer's Mill, Woodfibre, British Columbia
- 4
 Material when it passes the first unloading flange; further,
 Buyer assumes full responsibility for handling, storage,
 treatment, processing, consumption, possession, use, resale
 and disposal of Material after such unloading.
- 5 or Canadian

II. ADDITIONAL TERMS AND CONDITIONS.

1. General Description of the Material

Vanillin Black Liquor. See Exhibit B (Typical Analysis).

2. Quantity

Buyer's partial requirements for Material estimate to be that quantity of Material containing 4,000 tons of sodium per year; up to 13,000 barrels (546,000 U.S. gallons) of Material per delivery.

3. Price

\$240.00 (U.S.) per ton of sodium contained in Material delivered to Buyer.



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4. Price Revision

The price, point of delivery, service allowance, if any, and terms of payment herein specified may be revised as of the first day of any month after the date hereof (including the first such month), by written notice from Monsanto given not less than fifteen (15) days prior to the first day of any such month. Unless Buyer, by written notice received by Monsanto prior to the first day of such month, objects to the proposed revision, such revision shall become effective on the first day of such month. Buyer's written objection to such proposed revision received by Monsanto prior to its effective date shall permit Buyer to purchase elsewhere the quantities due during the ensuing month if Buyer shall be able to purchase such quantities of Material of equal quality manufactured in the United States at a lower delivered price from responsible manufacturers thereof, and shall have furnished Monsanto with satisfactory written proof of bona fide offers by such manufacturers and Monsanto shall have elected not to meet such offers, in which event Monsanto shall be released from its obligation during such month. If Monsanto desires to revise the price, point of delivery, service allowance or terms of payment pursuant to this section but is restricted to any extent against so doing by reason of any governmental request, law, regulation, order or action, or if the price, point of delivery, service allowance or terms of payment then in effect under this Agreement are altered by reason of governmental request, law, regulation, order or action, Monsanto shall have the right to (a) terminate this Agreement by written notice to Buyer, (b) suspend deliveries for the duration of such restriction or alteration or (c) have apply to this Agreement (as of the effective date of such restriction or alteration) any price, point of delivery, service allowance or terms of payment governmentally acceptable. Any delivery suspended under this section shall be cancelled without liability, but this Agreement shall otherwise remain unaffected.

5. Price Protection

Should Buyer at any time any shipment is due under this Agreement be offered, in good faith, a lower delivered price on like goods manufactured in the United States of equal quality and specification, in like quantity as the shipment involved for the same use by a responsible manufacturer of such component not affiliated with Buyer and furnish Monsanto satisfactory written proof of same, Monsanto will, at its option, either (a) supply such shipment at the lower price or (b) permit Buyer to purchase such quantity at the lower delivered



price from the manufacturer making such offer. Any quantity so purchased by Buyer with Monsanto's permission shall be deducted from the total quantity of this Agreement.

6. Freight and Taxes

Buyer shall pay all Canadian Federal and Provincial governmental fees, duties, taxes and other charges applicable to the importation of Material into Canada. Any increase in freight rates paid by Monsanto on shipments covered by this Agreement and hereafter becoming effective and any tax or governmental charge or increase in same (excluding any franchise or income tax or other tax or charge based on income) (a) increasing the cost to Seller of producing, selling or delivering the Material or of procuring components used therein or (b) payable by Monsanto because of the production, sale or delivery of the Material, such as Sales Tax, Use Tax, Retailer's Occupational Tax, Gross Receipts Tax, Value Added Tax, may, at Monsanto's option, be added to the price herein specified.

7. Shipments

The quantity shipped in any Agreement month may be limited by Monsanto to either (a) the average of the monthly quantities purchased by Buyer for the preceding Agreement months or (b) the maximum quantity covered by this Agreement divided by the number of months in the period of this Agreement (provided, however, that if different quantities apply to different time periods within the period of this Agreement, Monsanto may limit shipments based upon the current maximum quantity for the applicable time period under this Agreement divided by the number of months in such time period). Any quantity not shipped as a result of any such limitation shall be deducted from the total quantity of this Agreement. Monsanto shall not be bound to tender delivery of any quantities for which Buyer has not given shipping instructions.

8. Excuse of Performance

(a) Deliveries may be suspended by either party in the event of: Act of God, war, riot, fire, explosion, accident, flood, sabotage; lack of adequate fuel, power, raw materials, labor, containers or transportation facilities; compliance with governmental requests, laws, regulations, orders or actions; breakage or failure of machinery or apparatus; national defense requirements or any other event, whether or not of the class or kind enumerated herein, beyond the reasonable control of such party; or in the event of labor trouble, strike, lockout or injunction (provided that neither party shall be required to



settle a labor dispute against its own best judgment); which event makes impracticable the manufacture, transportation, acceptance or use of a shipment of the Material or of a component on which the manufacture of the Material is dependent.

- (b) If Monsanto determines that its ability to supply the total demand for the Material, or obtain any or a sufficient quantity of any component used directly or indirectly in the manufacture of the Material is hindered, limited or made impracticable, Monsanto may allocate its available supply of the Material or such component (without obligation to acquire other supplies of any such Material or components) among itself and its purchasers on such basis as Seller determines to be equitable without liability for any failure of performance which may result therefrom.
- (c) Deliveries suspended or not made by reason of this section shall be cancelled without liability, but this Agreement shall otherwise remain unaffected.

9. Handling Precautions

Handle in accordance with good industrial hygiene and safety practice.

Protective equipment:

Eyes: Chemical goggles

Skin: Rubber gloves and long sleeved clothing

In case of contact with skin, remove contaminated clothing and wash exposed surface with large volumes of water. If irritation persists, consult a physician.

In case of contact with eyes, flush immediately with large volumes of water. If irritation persists, consult a physician.

10. Entire Agreement

This Agreement immediately cancels and supersedes any prior agreement for sale of Material to Buyer by Monsanto. Each party shall fulfill any obligations which have accrued prior to the date of this Agreement.



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13. Address for Notices

Monsanto: Monsanto Company General Manager, Specialty Chemicals 800 N. Lindbergh Blvd. St. Louis, Missouri 63167

Buyer:

and

Plant Manager Monsanto 9229 E. Marginal Way South P. O. Box 80963 Seattle, Washington 98108





EXHIBIT B

Vanillin Black Liquor (VBL)

Typical Analysis

Calcium (ppm)	40
Soluble solids (weight %)	20
Sodium (weight %)	5
Sulphur (weight %)	1.5
ph	12.0
Specific Gravity at 68°F	1.15
Density at 68°F (pounds/gallon)	9.6
BTUs/pound dry solids	3,540

Monsanto will provide Buyer with an analysis of sodium and sulphur content of each shipment of VBL prior to or at the time of receipt by Buyer. The analysis of other components of VBL will be provided upon written request by monthly composite. See Standard Test Procedures attached hereto as Exhibit C.

MONSANTO INDUSTRIAL CHEMICAL COMPANY SPECIALTY CHEMICALS DIVISION SEATTLE PLANT

STANDARD PROCEDURE Number SRM-10A				
Department <u>Vanillin Laboratory</u> Page <u>1</u> of <u>1</u>				
Title Percent Solids Determination				
Date issued 11/24/74 Reviewed by BF				
Scope				
This procedure outlines the determination of Total, Soluble and Insoluble Solids.				
Equipments				
1. Analytical Balance 2. Drying oven mechanical convection 135°C 3. Aluminum pans 4. Centrifuge 5. Centrifuge tubes 6. Disposal droppers				
Determination				
 Weigh accurately approximately 2.0 gm. of well shaken sample into weighing pan. Place in oven at 135°C for 16 hours. Cool to room temperature and reweigh. Calculate the %Total solids from the weight of the residue divided by the total sample weight and time 100. 				
5. Repeat the same procedure for Soluble solids using centrifuged sample.				
6. Percent of Insoluble solids will be the difference between the Total solids and the Soluble solids.				

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Return to Office
of the Secretary



MONSANTO INDUSTRIAL CHEMICALS COMPANY SPECIALTY CHEMICALS DIVISION SEATTLE PLANT

STANDARD PROCEDURE

Number: SV-42

Revised 12/7/77

Department: Vanillin Laboratory Page 1 of 2

Pitle: SODIUM CONCENTRATION IN VANILLIN BLACK LIQUOR, AUTOCLAVE,

AND CONTINUOUS CLAVE SAMPLES BY ATOMIC ABSORPTION

SPECTROSCOPY

Equipment: 1. Instrumentation Laboratory, Inc. IL 751 Atomic Absorption/Atomic Emission Spectrophotometer

Reagents: 1. Certified 1000 ppm Sodium Standard

2. Deionized water

Instrument Operating Conditions:

- 1. Spectral Source: Sodium hollow cathode lamp
- 2. Lamp current: 6 8 mA
- 3. Wavelength: 330.2 nm
- 4. Slit Width: 0.160 nm; Bandpass: 0.5 nm
- 5. Burner Head: IL 43005-02 long path, single slot, air/acetylene (slot parallel to light beam)
- 6. Flame: Oxidizing, fuel-lean, blue
- 7. Fuel: Acetylene, welding grade (tank pressure: 15 psig; flow: about 4 5 SCFH)
- 8. Oxidant: Air, supplied by compressor (delivery pressure: 40 psig; flow: about 17 SCFH)
- 9. Aspiration rate: about 6 ml/minute
- 10. Scale expansion factor: 1.00

Procedure: 1. Centrifuge sample for five minutes at about 2000 rpm.

2. Dilute 2 ml of accurately weighed sample to 100.0 ml with deionized water. Further dilute 10.0 ml of this solution to 100.0 ml.



MONSANTO INDUSTRIAL CHEMICALS COMPANY SPECIALTY CHEMICALS DIVISION SEATTLE PLANT

STANDARD PROCEDURE

Number: SV-42

Revised 12/7/77

Department: Vanillin Laboratory

Page 2 of 2

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SODIUM CONCENTRATION IN VANILLIN BLACK LIQUOR, AUTOCLAVE,

AND CONTINUOUS CLAVE SAMPLES BY ATOMIC ABSORPTION

SPECTROSCOPY

Procedure: (Continued)

- 3. Prepare standards corresponding to 100, 120, and 150 ppm by diluting certified sodium standard with deionized water. Always prepare standard solutions such that their concentrations bracket those of the sample solutions.
- 4. Warm up lamp and burner head for about five minutes. With instrument in atomic absorption mode, optimize operating parameters.
- 5. Zero the instrument using deionized water.
- 6. Run standards and plot curve using calibration program.
- 7. Run sample and read out concentration using statistics program.

LOUSANTO INDUSTRIAL CHEHICALS COMPANY Detergent and Fine Chemicals Division SEATTLL PLANT

of the Secretary

STANDARD PROCEDURE

	•	Number SV-70
Department_	Vanillin Laboratory	3 Sheets: Page 1
Title	Total Sulfur in Vanillin Black	Liquor

Date issued October 6, 1976

Scope .

Total sulfur in VBL is determined by oxidation in an oxygen flask combustion apparatus followed by titration with barium perchlorate using an azo dye indicator

Equipment

- Thomas-Ogg combustion flask, 1000 ml, complete with stopper, platinum carrier, and pinch clamp (A. H. Thomas Co., 6514-F20) 1.
- Thomas-Ogg oxygen flask infrared igniter (A. H. Thomas Co., 2. 6516-G10)
- Sample wrappers, black (A. H. Thomas Co., 6514-F70)
- 4. Oxygen cylinder, industrial
- 5. Buret, 25 or 50 ml
- Mortar and pestle 6.

Reagents

- Hydrogen peroxide solution, 30% Bromphenol blue solution, 0.1% aqueous 2.
- Nitric acid solution, approximately 1 N 3.
- Isopropyl alcohol, reagent grade
- 5. Sulfonazo III solution, 0.1% aqueous
- Standard barium perchlorate titrant, approximately 0.01 M in 6. (1:1) isopropyl alcohol/water (3.36 g/l)
- 7. Standard sulfuric acid solution, 0.02 N

Standardization

- Transfer 10.0 ml standard 0.02 \underline{N} sulfuric acid solution to a 1. 250-ml conical flask followed by 5 ml water
- 2. Add 1 drop bromphenol blue solution, 15 ml isopropyl alcohol, and 5 drops Sulfonazo III solution
- Titrate with standard barium perchlorate solution to a blue endpoint (no trace of violet) persisting for 30 seconds or more (Note 1)
- Perform a blank determination in a similar manner, leaving out the sulfuric acid and adding 3 drops 1 N nitric acid solution following the bromphenol blue; add isopropyl alcohol, and Sulfonazo III



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Standardization - Continued .

5. mg S/ml Ba(ClO₄)₂ = $\frac{(N_{H_2SO_4})(10)(16.03)}{A-B}$

where A = volume titrant required for H_2SO_4 (m1) B = volume titrant required for blank (m1)

Procedure

- 1. Dry a centrifuged sample of VBL (about 50 g) in a 135° oven for at least 16 hours. Cool in desiccator and weigh. Percent soluble solids = weight residue ÷ weight sample x 100°
- Using a mortar and pestle, grind the dry sample to a fine powder
- 3. Transfer about 40 mg (Note 2) to a tared sample wrapper, weigh accurately, and wrap sample carefully, leaving about 1/2 inch of wrapper tail extending. Place in platinum sample carrier
- 4. Add 5 ml water and 5 drops hydrogen peroxide solution to the combustion flask. It is not necessary that the flask be dry; however, it is of extreme importance that the flask be free of volatile, flammable solvents, including isopropyl alcohol
- 5. Introduce free-flowing oxygen into the flask for about 1 minute
- 6. Clamp stopper holding sample carrier onto flask. Following manufacturer's directions, position flask in infrared igniter, and close and latch door
- 7. Close switch in line cord just long enough for paper target to ignite (Note 3). Open door only after ignition is complete
- 8. Shake flask for 1 minute, allow to stand for 20-30 minutes for white cloud to be absorbed, and shake for 1 more minute
- 9. Carefully rinse stopper with about 10 ml water, collecting rinsings in combustion flask
- 10. Add 1 drop bromphenol blue solution (blue color). Add 1 \underline{N} nitric acid solution to the yellow endpoint plus 3 drops in excess
- 11. Add 15 ml isopropyl alcohol followed by 5 drops Sulfonazo III solution
- 12. Titrate with 0.01 $\underline{\text{M}}$ barium perchlorate solution as in the standardization
- 13. $(C-B) (mg S/m1 Ba(C1O_4)_2)$ % Total S = weight sample (mg) x % soluble solids x 100 (as decimal)

where C = volume titrant required for sample B = volume titrant required for blank



Notes

- $H_2SO_4 + Ba(C1O_4)_2 \longrightarrow 2 HC1O_4 + Ba SO_4$ The presence of excess barium causes the azo dye to change from blue violet to blue. The titration will proceed slowly near the endpoint due to the slow rate of color
- 2. This amount of VBL should contain about 3 mg sulfur
- There should be no danger with oxygen flask combustions provided:
 - no traces of volatile, flammable solvents are present in the flask
 - no cracks are present in the flask
 - the stopper is firmly clamped onto the flask (Some pressure will develop when combustion begins, followed by a slight vacuum.)
 - the infrared light beam is directed onto the tail of the sample wrapper rather than the sample itself

References

- Mc Donald, K. L., TAPPI, 57 (3), 163 (1974) Personal communication, K. L. Mc Donald, Supervisor, Scientific Services, Mac Millan Bloedel Research LTD, Vancover, B.C.
- 3. USP, 18th Edition, United States Pharmacopeial Convention, Inc., Wash., D.C., 1970, p. 910



MONSANTO INDUSTRIAL CHEMICALS COMPANY SPECIALTY CHEMICALS DIVISION SEATTLE PLANT

STANDARD PROCEDURE

Number: SV-43

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Department: Vanillin Laboratory Page 1 of 2

Title: CALCIUM IN VANILLIN BLACK LIQUOR BY ATOMIC ABSORPTION

Equipment: 1. Instrumentation Laboratory, Inc. IL 751 Atomic Absorption/Atomic Emission Spectrophotometer

- 2. Reeve-Angel (Whatman) glass fiber filters,
 934 AH (or equivalent)
- 3. Suction flask

Reagents: 1. Certified 1000 ppm Calcium Standard

- 2. Deionized water
- 3. Stock solution of 5% Lanthanum in 25% V/V Hydro-chloric acid. (58.65 g of La₂O₃ plus 250 ml concentrated HCl diluted to 1000 ml with deionized water)

Instrument Operating Conditions:

- 1. Spectral Source: Calcium hollow cathode lamp
- 2. Lamp Current: 5 7 mA
- 3. Wavelength: 422.7 nm
- 4. Slit Width: 0.320 nm; Bandpass: 1 nm
- 5. Burner Head: IL 43005-02 long path, single slot, air/acetylene (slot parallel to light beam)
- 6. Flame: Stoichiometric, first appearance of yellow
- 7. Fuel: Acetylene, welding grade (tank pressure: 15 psig; flow: about 6 7 SCFH)
- 8. Oxidant: Air, supplied by compressor (delivery pressure: 40 psig; flow: about 17 SCFH)



MONSANTO INDUSTRIAL CHEMICALS COMPANY SPECIALTY CHEMICALS DIVISION SEATTLE PLANT

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Number: SV-43

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Title: CALCIUM IN VANILLIN BLACK LIQUOR BY ATOMIC ABSORPTION

Instrument Operating Conditions: (Continued)

9. Aspiration Rate: About 6 ml/minute

10. Scale Expansion Factor: 1.00

Procedure: 1. Centrifuge sample for five minutes at about 2000 rpm.

- 2. Filter through glass fiber filter using suction flask.
- 3. To 5.0 ml of centrifuged, filtered sample add 20 ml deionized water and 10 ml lanthanum stock solution. Dilute to 50.0 ml using deionized water.
- 4. Prepare standard solutions containing 5, 10, 15, and 20 ppm by diluting certified calcium standard with deionized water. Each of these solutions and the blank should contain 20 ml lanthanum stock solution per 100 ml.
- 5. Warm up lamp and burner head for about five minutes. With instrument in atomic absorption mode, optimize operating parameters.
- 6. Zero the instrument using the blank described in step 4, above.
- 7. Run standards and plot curve using calibration program.
- 8. Run sample and read out concentration using statistics program.

Calculation: ppm calcium in sample = ppm calcium in diluted sample X 10

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